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Thermoset Powder Coatings

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89.1 Introduction

In recent years, awareness of environmental conservation and pollution prevention has risen steadily. Governmental regulation and true concern for the environment have motivated chemists to modify all types of coatings to reduce environmental impact. The concept of environmental "friendliness" has dramatically changed the way that coatings are formulated.

Powder coatings are arguably the most environmentally "friendly" coatings. They do not contain solvents to be released as hazardous air pollutants (HAPs). Powder coatings release very low amounts of volatile organic compounds (VOCs) during the baking cycle. They produce virtually no waste material. And, they contain very few hazardous chemicals. (Note: The few hazardous chemicals that have found their way into powder coatings are decreasing as they are replaced with safer materials.)

89.1.1 Powder Coatings Defined

Powder coatings can be described as "ground up dry paint." They may also be referred to as pulverized plastics. They have properties in common with both materials. The polymeric resins that are used in producing powder coatings are similar in nature to those used in both paints and plastics. All three materials are composed of combinations of resin, pigment, filler, and various additive materials. They may be thermoplastic or thermosetting.

The primary difference between the three types of compounds is the molecular weight range of the polymers used as binders. Plastics use the highest molecular weight resins, paint the next highest, and powder coatings the lowest. Paint, of course, also contains various solvents to dissolve or dilute the coating for easy application.

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89.1.2 The First Powder Coatings

Powder coatings are relatively new finishes. The first application of a "dry paint" occurred in the 1940s.[1] At that time, pulverized thermoplastic formulations were flame sprayed over metallic substrates. The plastics melted in the flame, flowed onto the substrate, and cooled there to form a protective coating. This was a poor way to apply a protective barrier, as it was very wasteful. Material decomposed in the flame and was lost. Furthermore, there was very little control of film uniformity or thickness that was built up on the substrate. It was not uncommon for films to exceed 100 mils (~2500 microns).

In 1950, a British patent was issued to Schori Metallizing Process, Ltd., for a "hot-dip" coating process.[2] By this process, a heated metal part was dipped or rolled in a thermoplastic powder. Loss through decomposition was minimized, but film build was still extremely high. It was also difficult to maintain clean plant conditions, as handling the parts throughout the coating process produced high dust conditions.

The next innovation in the application of thermoplastic powders occurred in 1953. A patent was applied for by Erwin Gemmer of Knapsack-Griesheim AG (later acquired by Hoechst) for a "fluidized bed" coating process.[3] By this process, pulverized material is "floated" in a container by upward movement of airstreams that are forced to diffuse through a bed of porous material. The floating powder takes on properties of a fluid in that the particles "flow" in the airstreams much like molecules of boiling water bubbling in a saucepan. A German patent was issued for this process in 1955 and a U.S. patent in 1958.[4]

In fluidized bed application, the metal substrate is heated above the melting point of the powder and dipped into the fluidized material. Powder that touches the metal melts and adheres to the part. The part is then transported to the following stage of production by conveyor. The process is much cleaner than dipping or rolling, but film build is still very high.

89.1.3 Thermoset Beginnings

Up to this point, all powder coatings were thermoplastic in nature. Thermoset powder coatings were just beginning to be explored. The first trials were made with existing resins or slightly modified polymers. They had low glass transition points (T_g) and tended to sinter (cake) upon standing for any length of time.

In the early 1960s, two developments occurred almost simultaneously. The first development was the invention of storage stable thermoset powder coatings.[5] They were epoxies based on diglycidyl ethers of Bisphenol A. The second invention was the electrostatic spray gun, which was capable of applying a powder coating under controlled conditions.[6]

Electrostatic application of powder coatings is performed by pumping a stream of coating particles in an airstream past a high voltage cascade generator. The powder acquires an electrostatic charge and flows through the generated electrical field to a grounded metal substrate. The substrate is then transported to an oven where the coating melts, flows out, and is cured.

89.1.4 The Beginning of Growth

The electrostatic spray gun is probably the single most important invention in the rapid growth of thermoset powder coatings. With this equipment, virtually all of a powder coating product can be used in application, as oversprayed material can be reclaimed and used. Film build can be reduced to less than 5 mils (~125 microns). In fact, film thickness similar to that possible with liquid coatings can now be achieved (<1.0 mil or ~25 microns).

It has been reported that the world production of thermoset powder coatings in 1962 was around 16,000 lb (ca. 7.3 metric tons). All of these coatings were epoxies. By 1966, the volume had grown to 100,000 lb (ca. 45.4 metric tons).

During 1969, the first polyester resins designed for thermoset electrostatically applied powder coatings were developed.[7] They took a long time to cure and had caking problems. Nevertheless, powder coating production continued to increase.

By 1972, the world production of thermoset powder coatings had grown to 11.6 million pounds (ca. 5300 metric tons). That same year, the first carboxyl-terminated polyester resins were produced.[7] They

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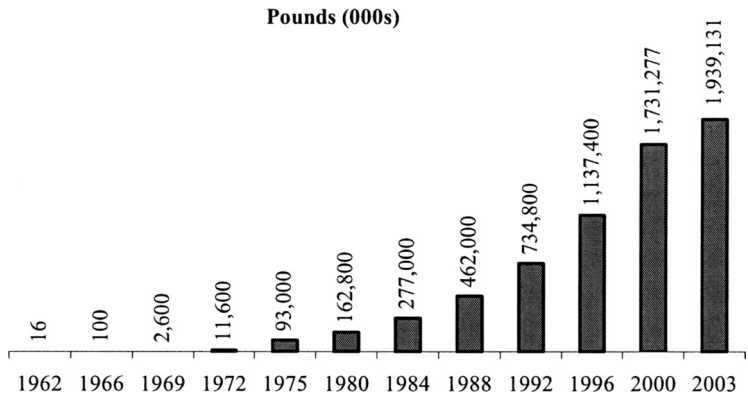

FIGURE 89.1 Worldwide growth of powder coating production for the years 1962 through 2003.

were the first polyester resins that eliminated the problem of outgassing due to reaction products forming bubbles and pinholes in the coating film.

Polyester resins with higher reactivity and, therefore, faster cure, were first introduced in 1974.[7] They started to gain in popularity in the next couple of years, and by 1983, the first trimellitic anhydride-free (TMA) polyesters were produced.[7] These were the earliest attempts to eliminate hazardous chemicals from powder coating formulae.

89.1.5 The World Discovers Powder Coatings

Powder production continued to grow at a fantastic rate. World production had grown from 162.8 million pounds (74,000 metric tons) in 1980 to 633 million pounds (ca. 290,000 metric tons) in 1990.[8]

About that time, it was discovered that TGIC (triglycidyl isocyanurate) could cause mutation in the chromosomes of lab mice. TGIC had been used as a cross-linker with certain carboxyl-terminated polyesters to produce extremely durable exterior coatings.

Immediately, chemists began to search for nonmutagenic alternatives. And, in 1990, the first non-TGIC cross-linkers for polyester resins were introduced.[9] The first was a β-hydroxyalkyl polyamide. It was followed in 1991 by tetramethoxymethyl glycoluril.

Even with the concern over mutagenic materials, powder production has continued to grow rapidly. Worldwide production of thermoset powder coatings for 1996 exceeded 1.13 billion pounds (over 1/2 million metric tons). The projected growth for the year 2002 is more than 1.8 billion pounds (>827,000 metric tons) worldwide and for 2003, it was over 1.9 billion pounds (> 880,000 metric tons) (see Figure 89.1).

89.2 Processing and Equipment

The process of producing a powder coating is somewhat more complex than that for other types of coatings. The equipment needs are more numerous. The production time is also somewhat longer. This section will describe each of the necessary pieces of equipment in the order that they are used during the process of making a powder coating.

89.2.1 Premixture

The first step in the manufacture of a powder coating is the premix stage. This is the most crucial phase of production. The various sizes of resin flake, pigment powder, etc., must be transformed into a homogenous blend of similar particle size before they enter the following stage — extrusion.

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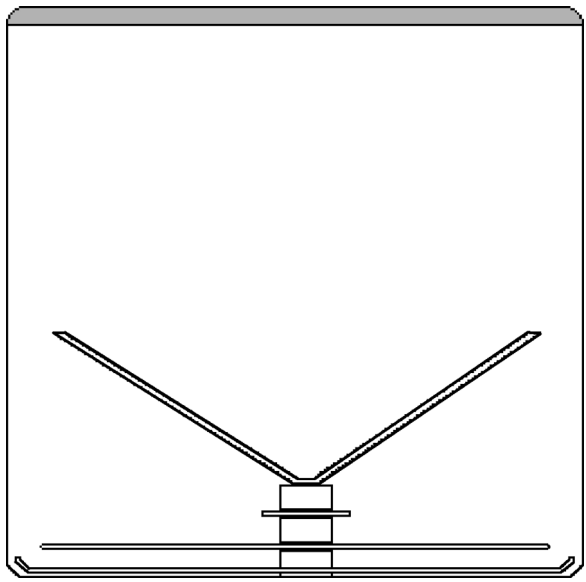

FIGURE 89.2 Generalized schematic of premix equipment.

Because the extruder is a simple melt mixer, and not a very efficient disperser, the premix stage must allow for the best possibility of good dispersion of all ingredients in the final product. Thus, premixture is performed in equipment with sufficient ability to break down the large resin flakes, thoroughly blend any liquid components, and disperse the smaller quantity constituents, such as additives.

89.2.1.1 Premixers

Most premix equipment consists of a mixing bowl with a locking cover. The equipment is usually fitted with a cooling jacket capable of being filled with running water or, in rare cases, cryogenic gasses. The mixing blades within the bowl may have various configurations. However, there are generally two or three blades placed at different heights and orientation to one another (Figure 89.2).

89.2.2 Extrusion

The second stage of powder coating manufacture is extrusion of the premix. The extruder is basically composed of a horizontally placed stainless steel tube or "barrel." Within the barrel are one or two "screws" that are slightly smaller than the barrel in diameter. The screws turn and move the premix forward through heated "mixing zones." The mixing zones have special attachments, called mixing paddles or blades. The paddles knead the melted premix and blend the various ingredients.

Contrary to what may be imagined, the extruder is not a very good disperser. The space between the screws and the barrel walls is relatively large and allows material to travel through without dispersing it thoroughly. Also, the coating mixture passes through the barrel fairly rapidly and does not have time to be dispersed efficiently.

89.2.2.1 Extruders

There are three basic types of extruder. The first type, the single screw extruder, is most often used in the plastics industry. It does not move material forward as rapidly as other types, and thermosetting coatings could react in the barrel and gel prematurely. Thermoplastic materials used in injection molding are commonly prepared in single screw extruders. However, there is one type of single screw extruder used in the thermosetting powder coatings industry. It has special attachments along the sides of the barrel that aid in the blending of the ingredients.

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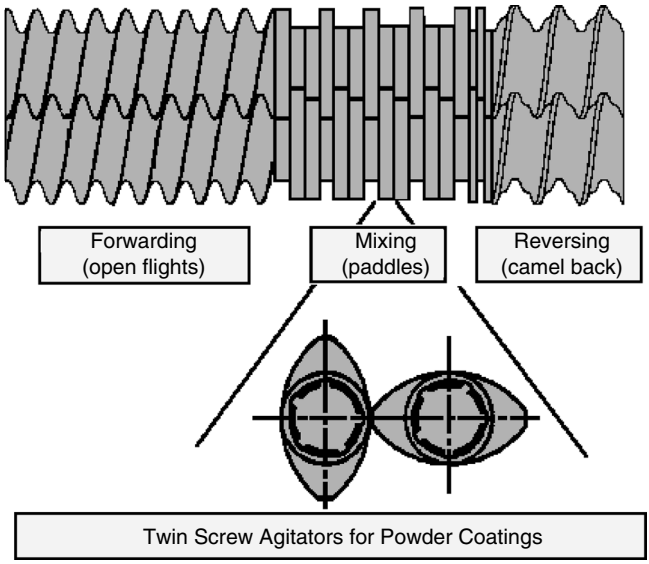

FIGURE 89.3 Typical configuration for twin screw corotating extruder.

The second type of extruder is the counter-rotating twin screw extruder. It is composed of two screws placed side-by-side and turning in opposite directions (i.e., one turns clockwise and the other counter-clockwise). This type is also usually used in the plastics industry.

The third kind of extruder, which is used in powder coating production, is the corotating twin screw extruder (see Figure 89.3). In this type of equipment, the two side-by-side screws turn in the same direction. This greatly increases throughput and decreases residence time. Reduced residence time is important in thermoset coatings, especially in the most reactive systems with short gel times.

89.2.3 Grinding

The grinding phase in the manufacture of a powder coating is important to its final performance. The average particle size and distribution are important in the application properties and final surface appearance of the coating.

Once a powder coating has been extruded, it passes through one or more stages to prepare it for grinding. The molten material is dropped onto chilled metal rollers, where it is pressed flat and cooled. The coating is then broken into flakes or granulated and fed to the grinder.

89.2.3.1 Grinders

There are three favored types of grinding equipment used in the powder coating industry. They are the impact or hammer mill, the air jet mill, and the air classifier mill.

The impact or hammer mill grinds the coating materials by throwing them or hitting them against a stationary screen or stator.[10] The particles are broken down until they are small enough to fit through the screen. The particles are then passed to various sieves or cyclonic separators for final particle size distribution adjustments.

The air jet mill is an extremely efficient grinder. Powder coatings are fed into a high volume and high velocity airstream. The particles circle the cylindrical grinding chamber striking other particles and breaking them into very fine powders. The fine material rises out of the chamber with the escaping air, through conduits, to separation devices that structure the particle size distribution.

The air classifier has elements of both of the previous types. There is an impact chamber that is supplemented with airstreams that rapidly break down the coating materials. This provides excellent

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control of particle size distribution, and the moving air tends to cool the powder, minimizing heat effects, such as impact fusion.

89.2.4 Sifting and Classifying

The final structuring of particle size distribution is accomplished by passing the ground materials through sieves or cyclonic separators. Sieves "scalp" the largest or smallest particles, which narrows the particle size distribution. By judicious use of sieves, the distribution and average particle size may be chosen for best application and surface properties.

Cyclonic separators or "classifiers" function by forcing powders in airstreams to circle conical chambers. The heaviest (largest) particles will fall to the bottom of the chamber and will be collected for use or dumping. The smallest particles will flow out through the top with escaping air, will flow through conduits, and will be saved or discarded as the need arises. By adjusting the volume and velocity of the airstream, a particle size distribution can be varied.

89.2.5 Application Equipment

There are three primary methods of applying powder coatings. The first uses a fluidized bed. As described earlier, the fluidized bed transmits a diffuse stream of air upward through the bottom of a container holding the coating to be applied. The airstream is kept at low enough pressure so that the powder rises into the air but is not expelled from the container. As air escapes past the floating particles, the surface of the powder mass appears to bubble or "percolate." The term "fluidized" refers to this fluid-like motion. The metal part to be coated is heated above the melting point of the coating and is "dipped" into the fluidized bed. Powder touching the part melts and adheres to the metal. The part is then transported to a curing oven.

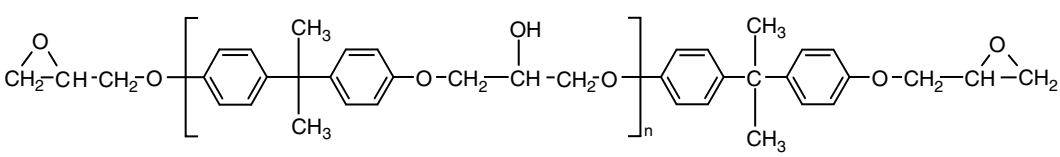
The second and third methods of application utilize electrostatic spray guns. There are two basic types of gun: corona-charged and "tribo"-charged. The corona-charged spray gun applies an electrostatic charge to the powder as the material flows through an electrical field generated at the gun tip of the gun. The tribo-charged gun has a polytetraflouroethylene (PTFE) barrel, and powder flowing through it acquires an electrical charge from friction. The charged powders are sprayed onto grounded metal parts, where they adhere through electromagnetic attraction. The parts are then transported to the curing oven.

89.3 Chemistry

The chemistry of powder coatings is not very different from that of liquid coatings. The main resin types used in thermosetting powder coatings are all familiar to the liquid coatings chemist. The epoxy, polyester, and acrylic polymers are well known in liquid coatings. However, the molecular weight and curing agents are somewhat different. From these three resin types, a number of different cross-linked systems are possible. The most common will be discussed here. Note that each subsection describing a cross-linking pair includes a common industry "nickname" in parentheses.

89.3.1 Epoxy Systems

The most common epoxy resins in use today are diglycidyl ethers of Bisphenol A made from reaction of epichlorohydrin with Bisphenol A. Diglycidyl ethers of Novolac resins also have significant commercial impact.



Diglyceride Ether of Bisphenol A Resin

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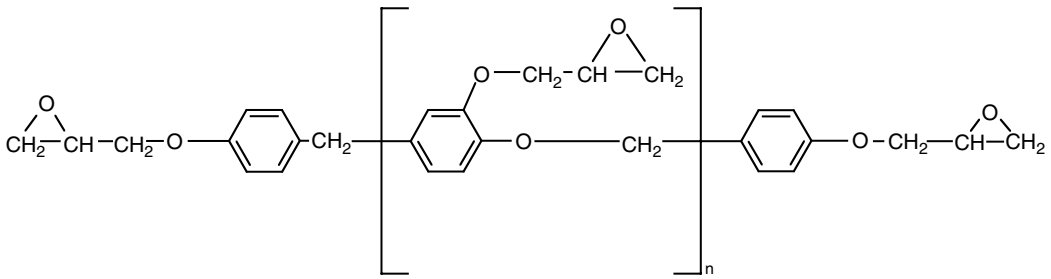
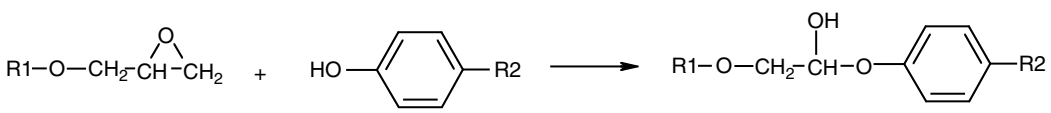
The epoxy groups terminating each molecule react with acidic or basic curing agents. The three most common are the phenols, dicyandiamides (DICY), and carboxylic acids, including carboxy-terminated polyesters. Various acids, anhydrides, amines, and imidazoles are also used as cross-linkers with epoxy resins.



Diglycidyl Ether of Novolac Resin

89.3.1.1 Epoxy-Phenols (Phenolic)

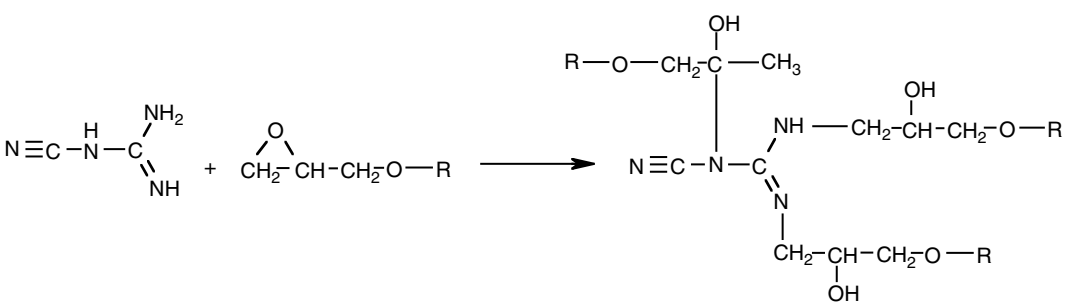
The curing of epoxy resins with phenols results in the opening of the epoxide ring and the formation of a hydroxyl group, at either the primary or secondary position. The hydroxyl group is available for reaction in the cross-linking of the resin. The aromatic ring attaches to the unreacted carbon of the epoxide.



Epoxy Resin Phenolic Crosslink Intermediate

89.3.1.2 Epoxy-Dicyandiamide (DICY)

DICY cured epoxy coatings react in a similar manner to that of the previous type, where nitrogen-bearing groups replace the aromatic ring. All four functional groups will react with the epoxide, acting as a primary or secondary amine.



DICY Epoxy Resin Crosslink Intermediate

89.3.1.3 Epoxy–Polyester (Hybrid)

Epoxy resins react with carboxy-functional polyesters in the same way as carboxylic acids. The hydroxyl portion of the acid group reacts with the epoxide. The rest of the reaction follows as we have seen in the previous examples. Because both reactants are considered primary resins, the system is referred to as a "hybrid."

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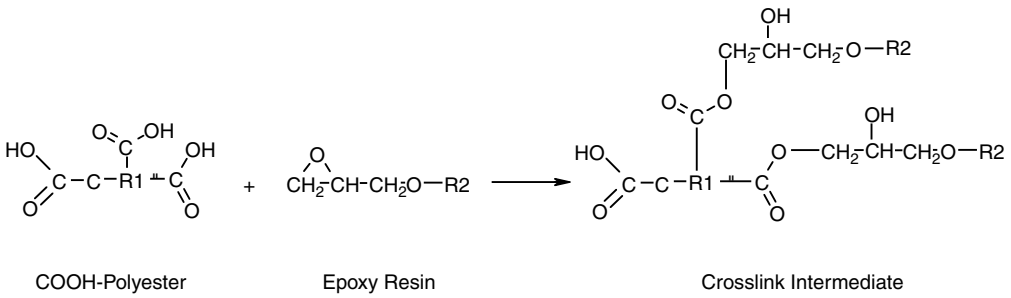
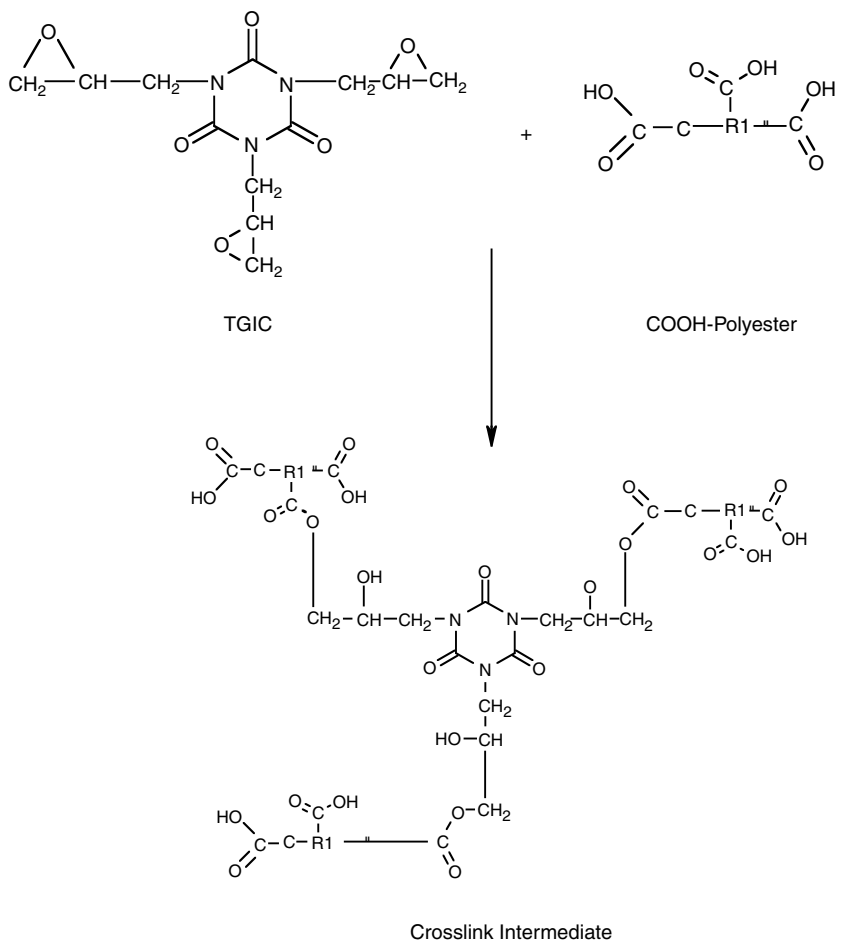


89.3.2 Polyester Systems

There are two types of polyester resins used in thermoset powder coatings. They are carboxy- and hydroxy-functional. There are several different curing agents used with each. We will examine the four most common systems.

89.3.2.1 Polyester-Triglycidyl Isocyanurate (TGIC or Polyester)

Triglycidylisocyanurate (TGIC) is a heterocyclic tri-epoxy curing agent. As such, it reacts with carboxy-functional polyester resins similar to those used in hybrid coating systems. The major difference is in the trifunctionality. One mole of TGIC will react with three moles of polyester resin. The cross-link network is also more complex because of the extra reactive sites available.



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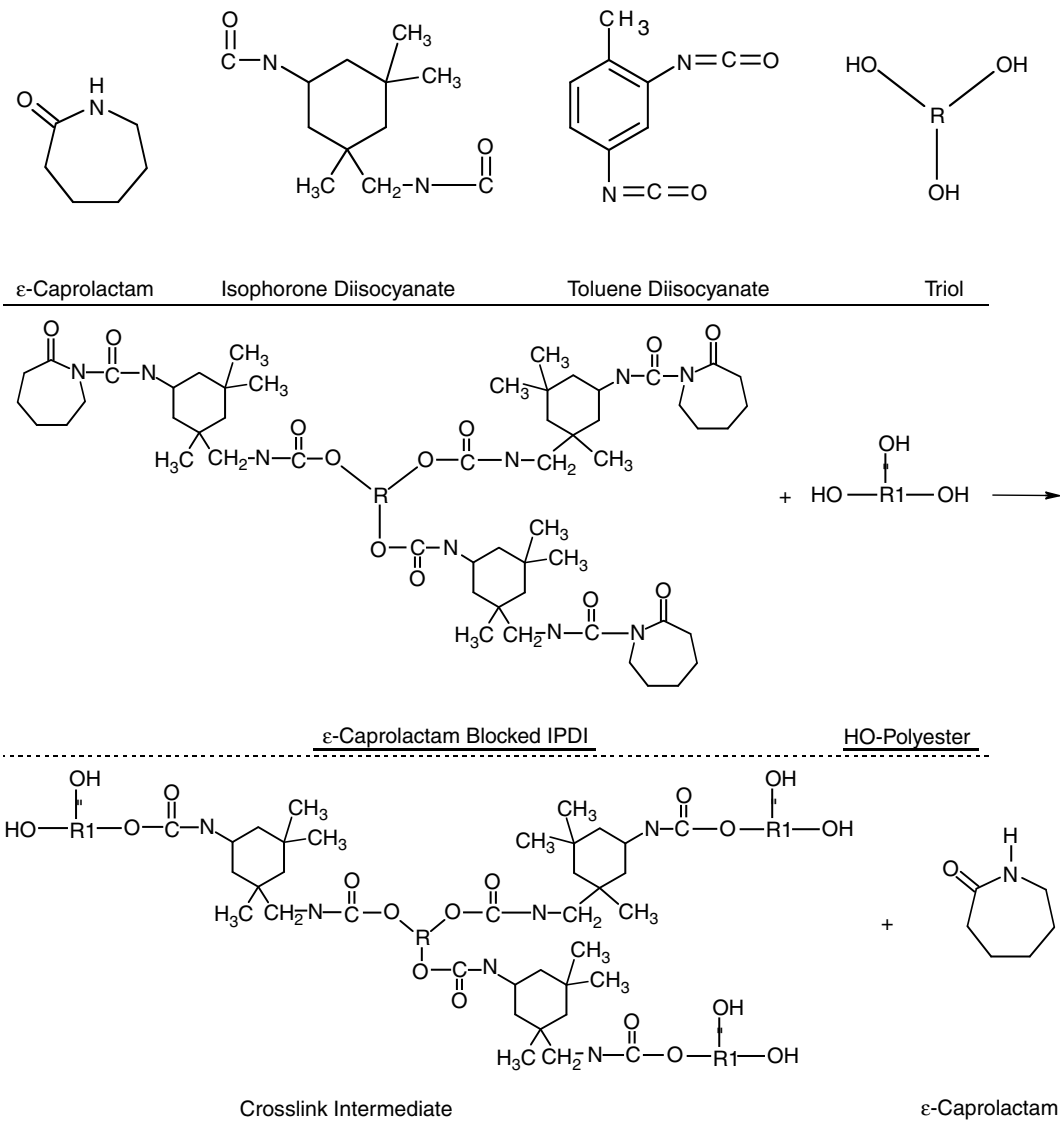
89.3.2.2 Polyester–Isocyanate (Polyurethane)

Polyester resins used to produce urethanes have hydroxyl functionality. They react at the carbon–nitrogen bonds in an isocyanate. The two isocyanates most used in powder coatings are isophorone diisocyanate (IPDI) and toluene diisocyanate (TDI). However, TDI melts at room temperature, and IPDI is a liquid. This would cause any powder coating in which they were used to have poor package stability. The coating would sinter (cake) into a solid very quickly. Furthermore, they would react with the polyester and gel during the heat of extrusion, again making them unusable.

Consequently, isocyanates are reacted with triol materials to give them higher melting points and better package stability when compounded in powder coatings. Then they are blocked, most commonly with (epsilon) ε-caprolactam, to prevent them from reacting with the polyester until they unblock naturally at curing temperatures in the oven.



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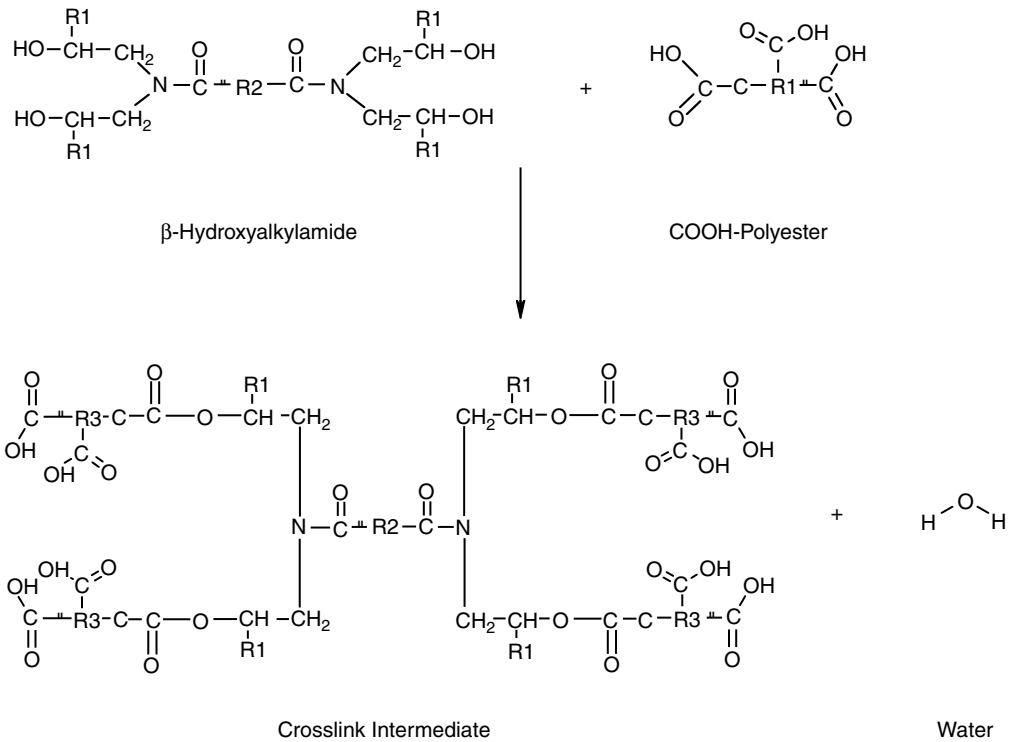
89.3.2.3 Polyester–Non-TGIC (β-Hydroxyalkylamide, Tetramethoxymethyl Glycoluril)

As we discussed earlier, TGIC was found to have mutagenic properties. While some controversy still remains as to its hazardous nature, many coatings companies and regulatory bodies have taken a conservative stance and limited its use. There have been regulations enacted that require warnings to be placed on labels of coatings that contain TGIC. Coatings companies have also turned to curing agents that do not have these types of hazards associated with them.

The two types that are used most often are the β-hydroxyalkylamides and tetramethoxymethyl glycoluril. The hydroxy-amides are tetrafunctional, which makes them highly reactive at curing temperatures. Their major drawback is that they release water from the reaction that must escape the curing film. This requires further formulation to insure defect-free films. The glycoluril is also tetrafunctional and releases a VOC, methanol, as part of the reaction process.



89.3.3 Acrylic Systems

There are two primary acrylic systems: those based on hydroxy-functional acrylic resins and those using epoxy- or glycidyl-functional polymers. Carboxy-functional materials have been produced. However, they have not made much progress into the industry.

89.3.3.1 Acrylic–Isocyanate (Acrylic–Urethane)

Acrylic–urethanes are formed in exactly the same way as their polyester counterparts. The acrylic resins are linear instead of aromatic. However, they use hydroxy-functionality and blocked-isocyanates to form the urethane bonds.

89.3.3.2 Acrylic–Diacid (Glycidyl–Acrylic)

Epoxy- (glycidyl-) functional acrylic resins can be compared to the hybrid systems discussed earlier. They are generally reacted with dicarboxylic acids or anhydrides. The most common cross-linker is 1,12-duodecanoic acid (1,12-dodecane dioic acid).

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89.4 Formulation

89.4.1 Resin Systems

Formulation of thermoset powder coatings is much the same as that for liquid coatings used for similar purposes. A coating may be chosen for functional or decorative purposes. All of the coating types used in powder coatings can offer decorative options. Nonetheless, it is function that dictates system choice. The resin chemistry must be chosen to suit service needs. Various pigments, fillers, and additive materials are then included to enhance decorative or functional requirements.

There are two primary functions for any protective coating. They are chemical protection and exterior durability. As with many coating properties, these two tend to be in opposition. The best systems for chemical protection are usually the poorest for exterior durability.

Epoxy powder coating systems deliver the best chemical and corrosion resistance. However, they have the least effective exterior durability. The double bonds in the aromatic rings are easily broken by the ultraviolet (UV) light from the sun. Glossy finishes will go flat with as little as 6 months of exposure, with film degradation following soon thereafter.

Urethane systems are also very good for chemical resistance, and they have fairly good exterior durability, as well. Hybrids offer good chemical resistance, however, the polyester component makes them less effective. They are also poor in relation to exterior exposure due to the epoxy portion of the cross-link network.

Acrylic and TGIC powder coatings provide the best exterior durability. Some systems can survive for up to 20 years of exposure. They offer fair to good chemical resistance. Urethane systems seem to be the best compromise between chemical and exposure properties. As mentioned, they are very good in both areas.

89.4.2 Pigments and Fillers

Most pigments and fillers used in liquid coatings are suitable for use in powder coatings.[11] There are only a few special requirements for use. They must be sufficiently heat stable so they withstand the heat of extrusion and curing without degradation or color change. Normally, the heat of extrusion is 125°C or less for a minute or two. The heat of cure is usually 160 to 200°C for 10 to 20 min.

Second, they must be insoluble and nonreactive in the resin system. Blooming and color shift are the most common results from pigments that are partially soluble or reactive with the binder. Some epoxy system curing agents are especially susceptible to reaction with pigments.

89.4.3 Additives[12]

89.4.3.1 Flow and Leveling

Flow and leveling agents are designed to minimize surface defects such as craters, pinholes, and orange peel. The mechanism of their function alters the surface tension and rheology of the coating. The likelihood of a smooth defect-free film is improved by reducing one (or both) of these properties in a coating.

Most flow and leveling agents are liquids. Many are blended with inert inorganic materials to offer them in a conveniently solid form. The chemistries of these are usually polyacrylates or polysiloxanes. A few new flow agents are available, however, in solid organic form.

89.4.3.2 Debubbling (Degassing)

The most common debubbling agent is benzoin (2-hydroxy-1,2-diphenyl ethanone). It is used to keep the surface of a curing film open long enough to allow for entrained air and evolved gasses to escape. Trapped air and gas bubbles are cause for premature failure of coating films, because they make the coating brittle. The one drawback to the use of benzoin is its tendency to cause yellowing in lighter colors. A number of new advances have entered the market in an attempt to match the efficiency of benzoin without the challenge of yellowing.

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89.4.3.3 UV Inhibitors

Various UV light inhibitors are available to aid coating resistance to degradation by the sun's rays. The most common are hindered amines, phosphites, sulfates, and phenolics. Most will have some positive effect on any coating's UV resistance. However, each system will require testing to determine the best combination of inhibitors. Some systems, like epoxies and hybrids, will not develop any substantial UV resistance due to their aromatic nature.

89.4.3.4 Catalysts

Catalysts or accelerators are used to reduce the reaction time or curing temperature of the resin and cross-linker. They allow for faster production time by shortening the gel or "set" time of the thermosetting coating. Energy can be conserved, because full cure may be attained at lower oven temperatures. The most common catalysts are thiazoles (used in polyesters), phosphines and ammonium halides (used in epoxies), and thiocarbamates (used in urethanes).

89.5 End Uses

The following table details some of the applications currently using the chemistries discussed in this chapter.

Type	Typical Applications
Epoxy	Shelving, transformer cases, primers, bathroom fixtures, refrigerator racks, sweepers, sewing machines, power tools, room air conditioners, office furniture, instrument cases, garden tools, kitchen furniture, fire extinguishers, toys, refrigerator liners, dryer drums, microwave ovens, mixers and blenders, fertilizers spreaders, screening, oil filters, automobile springs, hospital equipment, bus seat frames, business machines, glass bottles
Hybrids	Tool boxes, farm equipment, electrical control boxes, hot water heaters, hot water radiators, primer/surfacers, grain storage bins, transformer covers, 01.1 filters and air cleaners, air conditioner housings, fire extinguishers, toys, screening wire, power tools, shelving, office furniture
Urethane	Fluorescent light fixtures, steel and aluminum wheels, patio furniture, playground equipment, fence fittings, chrome wheels and trim, garden tractors, range side panels and broiler, ornamental iron, air conditioner cabinets, restaurant furniture supports, transformer cases
TGIC	Irrigation pipe and fixtures, outdoor furniture, air conditioning units, steel and aluminum, wheels, wire fencing, fence poles and fittings, farm equipment, aluminum extrusions, transformers
Acrylic	Range side panels, refrigerator cabinets and doors, washing machine parts, dishwasher exterior, aluminum extrusions, microwave ovens, garden tractors, automotive trim coating

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